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**DIVISION OF ENGINEERING SERVICES**  
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## METHOD OF TESTING SOILS AND WATERS FOR CHLORIDE CONTENT

**CAUTION:** Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read "**SAFETY AND HEALTH**" in Part 3 of this method. It is the responsibility of the user of this method to consult and use departmental safety and health practices and determine the applicability of regulatory limitations before any testing is performed.

### OVERVIEW

This method describes test procedures for determination of the Chloride content of soils and waters. These results are used in determining the corrosive nature of the environment for concrete structures, as well as for other purposes. This test method is divided into the following parts:

1. Chloride content of waters
2. Chloride content of Soils
3. Safety and Health

### PART 1. CHLORIDE CONTENT OF WATERS

#### A. SCOPE

This method is used to determine the chloride content of waters.

#### B. REAGENTS AND MATERIALS

Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

1. Preparing Eluent – Dissolve 21.0 g of Sodium Bicarbonate,  $\text{NaHCO}_3$  in deionized water and dilute to 500 mL

volume. Dissolve 26.5 g of Sodium Carbonate,  $\text{Na}_2\text{CO}_3$  in deionized water and dilute to 500 mL volume. Transfer each solution to a separate polypropylene bottle. Prepare eluent solution by adding 2 mL of  $\text{NaHCO}_3$ , 7 mL of  $\text{Na}_2\text{CO}_3$  solutions, and dilute to 1000 mL with deionized water.

2. Standard Sulfate solution, 1000 ppm: Dissolve 1.8145 g of  $\text{K}_2\text{SO}_4$  in deionized water and dilute to 1000 mL.
3. Standard Chloride solution, 1000 ppm: Dissolve 1.6485 g of  $\text{NaCl}$  (dried at  $140^\circ\text{C}$ ) in deionized water and dilute to 1000 mL.
4. Volumetric/ automatic pipettes.
5. Volumetric flasks: 100 – 1000 mL
6. Erlenmeyer flasks: 500 mL and stopper #10
7. Poly vial: 5 mL vials and caps for use in autosampler
8. Acrodisc CR 0.5-1.0 inch syringe filter with 0.45 micron PTFE membrane
9. Disposable 10 mL syringes

### C. ANALYTICAL INSTRUMENT AND EQUIPMENT

Ion Chromatograph (IC) – The Dionex DX 120 model has been found to be satisfactory for this method. Any comparable instrument can be used as well.

### D. TEST PROCEDURE

#### 1. Calibration Curve:

Prepare a mixture of sulfate and chloride standards at concentrations of 5, 10, 20, 50, 100, and 200 ppm. In six 100 mL volumetric flasks, pipette, 0.5, 1, 2, 5, 10, and 20 mL each sulfate and chloride solution at 1000 ppm (Part 1, Section B.2 and 3), dilute to 100 mL with deionized water. Transfer each standard into a polypropylene bottle. Pipette 5 mL of each standard into a sample vial to run on the Ion Chromatograph.

**Note:** The Ion Chromatograph is set up to analyze chloride and sulfate from one run.

2. Blank: Pipette 5 mL of deionized water into a sample vial and cap. Blank is run at the beginning of every run to determine possible contamination.
3. Water samples: Pipette 5 mL into a sample vial and cap. Samples should be free of particles; otherwise, syringe filters must be used to filter each sample.
4. Prepare a program on the Ion Chromatograph by running a blank, calibration curve standards, check standard, samples, and shutdown mode at the end of the run. Samples with high concentrations of chlorides require dilution.

### E. CALCULATION

$$A_s = \text{Function } (R_s) * V_c / V_s * D_s$$

Where

$A_s$ : is the amount of the analyte in the sample

Function: is a form of the current calibration curve function

$R_s$ : is the response of the analyte in the sample

$V_c$ : is the volume of standard injected

$V_s$ : is the volume of sample injected from the method or schedule

$D_s$ : is the dilution factor from the method or schedule for the sample

## PART 2. CHLORIDE CONTENT OF SOILS

### A. SCOPE

This method describes the test procedure for determination of the water-soluble chloride content of soils.

### B. REAGENTS AND MATERIALS

Refer to Part 1, Section B for these items.

### C. EQUIPMENT

Refer to Part 1, Section C for analytical instrument and equipment.

### D. TEST PROCEDURE

1. Prepare calibration curve as in Part 1, Section (D.1).
2. Weigh 100 g of soil and place it in a 500 mL Erlenmeyer flask. Add 300 mL of deionized water, place a stopper on the flask, and shake vigorously for 15 minutes. Centrifuge

the sample, then filter the sample or let the sample settle overnight.

3. Pipette 5 mL of sample into a sample vial and cap. All samples must be filtered using syringe filters to ensure they are free of particulates.
4. Prepare a run program for the Ion Chromatograph with a blank, calibration curve standards, check standard, samples, and shutdown mode at the end of the program. Dilution factor of three is entered to compensate for the 3 to 1 extraction of soil.
5. It will be necessary to make dilutions on samples with sulfate content higher than the range of the calibration curve.
6. Check the reliability of the calibration curve by running a check standard for every 10 unknown samples.

#### **E. CALCULATION**

Calculation of the chloride in soil samples is the same as in Part 1, Section E.

#### **F. REPORTING RESULTS**

Record all procedures and data in a bound, numbered, laboratory book, and record on appropriate forms as required.

#### **G. ALTERNATE PROCEDURES**

Another method that may be used is "Standard Methods", 17<sup>th</sup> Edition, 1989, "Potentiometric Method."

### **PART 3. SAFETY AND HEALTH**

Prior to handling, testing or disposing of any of waste materials, testers are required to read: Part A (Section 5.0), Part B (Sections: 5.0, 6.0, 10.0, and 12.0) and Part C (Section 1.0) of the Caltrans Laboratory Safety Manual. These sections pertain to requirements for general safety principals,

standard operation procedures, protective apparel, disposal of materials and how to handle spills, accidents, emergencies, etc. Users of this method do so at their own risk.

#### **REFERENCES**

**Standard Methods for the Examination of Water and Wastewater, 17<sup>th</sup> Edition, 1989, APHA-AWWA-WPCF**  
**U.S. Environmental Protection Agency Manual (EPA), "Methods for Chemical Analysis of Water and Waste"**  
**ASTM Designation: D516**

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**(California Test 422 contains 3 pages)**